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SUMMARY

A guanidine-based vehicle was developed to deposit precious metal coatings on surfaces known to be difficult to coat. To demonstrate this method, a platinum coating was deposited on alumina fibers using a guanidine-platinum solution. X-ray diffraction confirmed that the only species present in the coating was platinum, and, that all of the carbon species had been removed upon heat treatment. SEM results showed that some porosity was present but that the coatings uniformly covered the fiber surface and adhered well to the fiber.

INTRODUCTION

The preparation of thin metallic films by the decomposition of metallo-organic solutions is a technology which has been known for many years; the first publications reporting on this technology appeared more than one hundred years ago. Although the technique has been known for a long time, its primary application has been in the decorative trade. The main emphasis is producing films of precious metals on ceramic and glass articles (ref. 1).

Almost all of the work prior to 1980 used metallo-organic compounds derived from resins or other natural products and are commonly called metal resinates. These resinates are suitable for most applications in the decorative trade. The variability of the chemistry is a result of the preparation from natural products, and is a major impediment to the extensive development of the technical applications of the metal-organic decomposition (MOD) processes. In recent years, MOD films have been produced from pure, well-characterized compounds and are now used in technical applications (ref. 2). For example, silver films are fabricated by thermally decomposing the MOD silver solutions on silicon for use as collector grids for photovoltaic cells. These collector grids required good adhesion, low contact resistance, low sheet resistance and long-term electrical stability. The fired films are 100 percent solder leach-resistant and have good definition and excellent long-term adhesion. This method of metallizing solar cells is inexpensive since less precious precursor material is required as compared to source targets needed for sputtering silver on silicon (ref. 3).

The conventional metallo-organic solutions have the advantage of producing film densities near the theoretical limit at low temperatures. However, adhesion to some ceramics is poor. Altering the viscosity of the solutions can alleviate the problems with adhesion. With more organics present in the solution, however, there is a greater tendency towards blistering.

A guanidine vehicle has been developed (ref. 4) that solves the adhesion and the blistering problems. This vehicle can be used to deposit precious metal films or metal oxide films on smooth, cylindrical surfaces and on smooth, flat metal surfaces. Guanidine is a strong base equal in strength to sodium hydroxide; thus, guanidine forms stable soaps with organic fatty acids that have high ionicity. This enables them to wet and to bond to the ceramic substrates. The advantage of these soaps is that they completely burn off and leave no residue when fired. The disadvantage of sodium-containing soaps is that they leave a sodium oxide ash upon firing. This ash can reduce the high-temperature strength of some ceramics.

For this study, single-crystal Al_2O_3 fibers were coated with a Pt-guanidine solution. This particular coating was chosen because of its ability to act as a sintering barrier for the fibers when placed in an oxide matrix. Al_2O_3 fibers were pulled through a melted guanidine-platinum solution in the guanidine 2-ethylhexanoate vehicle, air dried and subjected to a rapid thermal anneal to eliminate the organics and produce a porous Pt coating. The coating and firing procedures were repeated several times to increase the coating thickness on the fibers. Then, the fibers were annealed at 1000 °C to densify the Pt coating. X-ray diffraction and SEM were used to analyze the coatings.

Debye-Scherrer results indicated that only Pt peaks were present in the coatings. Thermal gravimetric analysis (TGA) of the Pt-guanidine solution confirmed that the organics were completely removed at 600 °C and that only some trace metals were evident. These trace metals were also present in the Pt-guanidine solution container walls.

Figures 1(a) and (b) show the surface and cross-section of one of the Pt-coated fibers after the anneal. Even though some porosity is still present in the coatings, the adhesion of the Pt coating is good. The coating is also fairly uniform over the length of the fiber (fig. 1(a)). Slower heating cycles were tried after each coating to reduce the porosity. However, as shown in figure 2, the coating was not as dense as those that were fired rapidly after each coating.

The coating thickness is dependent on the viscosity of the Pt-guanidine solution and on the number of applications. The coating and firing procedures for the fibers in this study were repeated four times, yielding approximately a 10 μm coating.

Although no chemical reactions were observed between the Pt and Al_2O_3 fiber, pitting on the surface of the fibers was observed by Jaskowiak (ref. 5) when the fibers were subjected to further heat treatments and when placed in an oxide matrix material. This has also been observed when other similar sol-gel coatings have been applied to these Al_2O_3 fibers (ref. 6). The pitting can be correlated back to impurities trapped at the surface by the coatings. These impurities, identified as Ca, Ti and other light metals, have also been observed using x-ray photoelectron spectroscopy (XPS) on the surface of the fibers before and after cleaning and are not necessarily from the Pt-guanidine solution or other chemical processes for coating the fibers. Jaskowiak (ref. 5) also observed that the Pt coating no longer adhered to the surface of the fiber above 1300 °C. However, the coating remained intact below 1300 °C.

It is possible to coat smooth, cylindrical surfaces with noble metals by means of a modified MOD process using a guanidine soap solution for the organic vehicle. Application of this process can range from thin films used as electric conductors in the microelectronics industry to additional coatings for composite materials. The Pt-guanidine solution utilized in this study adhered well to the Al_2O_3 fibers below 1300 °C. The coating was fairly uniform over the entire surface. Some porosity is present in the coatings, however, the Pt coating still provided the necessary barrier between the Al_2O_3 fiber and the oxide matrix for high temperature use.

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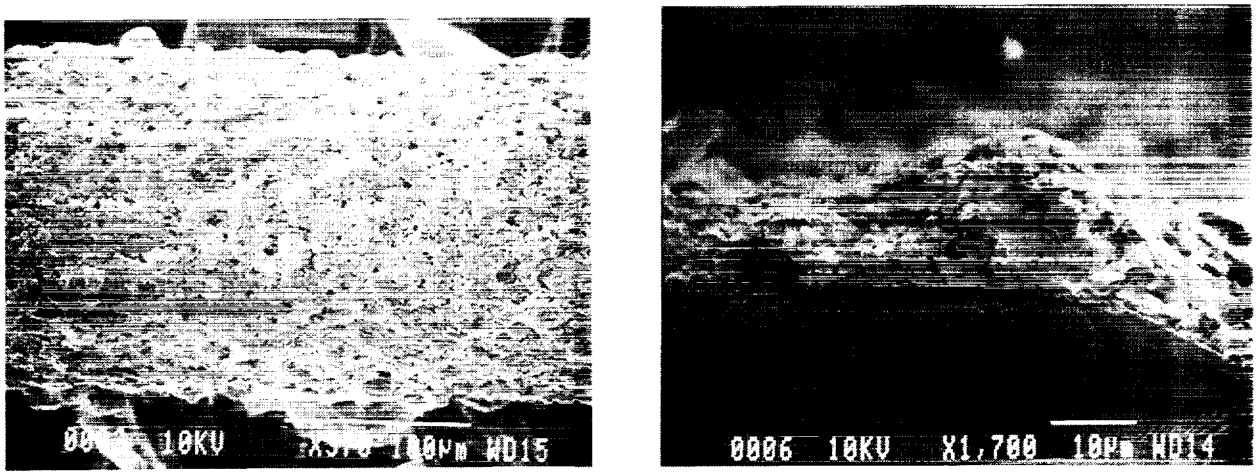

(a) Surface view. (b) Cross section.

Figure 1.—A platinum-coated single crystal alumina fiber after four coating and firing cycles and final anneal at 1000 °C.

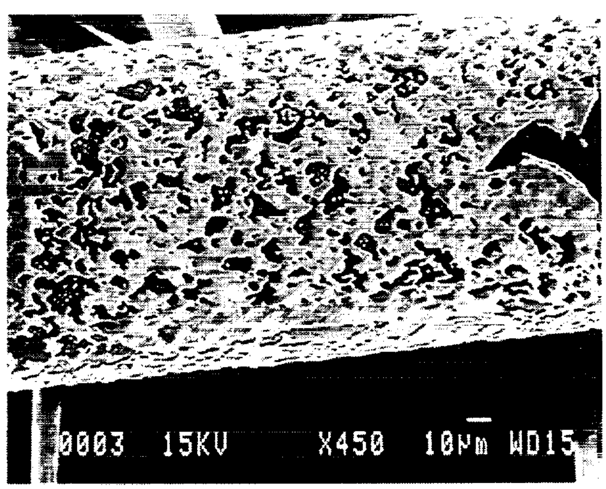

Figure 2.—The surface of a platinum-coated alumina fiber after 8 coating and slower firing cycles and a final anneal at 1000 °C.

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